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*Published in:*  
Data in Brief

*DOI:*  
[10.1016/j.dib.2017.08.053](https://doi.org/10.1016/j.dib.2017.08.053)

*Publication date:*  
2017

*Document version*  
Publisher's PDF, also known as Version of record

*Document license:*  
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*Citation for published version (APA):*  
Longo Martins, M., Eckert, J., Jacobsen, H., Carvalho, E. D. S., Ignazzi, R., de Araujo, D. R., Bellissent-Funel, M. C., Natali, F., Koza, M. M., Matic, A., de Paula, E., & Nunes Bordallo, H. (2017). Raman and Infrared spectroscopies and X-ray diffraction data on bupivacaine and ropivacaine complexed with 2-hydroxypropylcyclodextrin. *Data in Brief*, 15, 25-29. <https://doi.org/10.1016/j.dib.2017.08.053>



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Contents lists available at ScienceDirect

## Data in Brief

journal homepage: [www.elsevier.com/locate/dib](http://www.elsevier.com/locate/dib)

## Data Article

# Raman and Infrared spectroscopies and X-ray diffraction data on bupivacaine and ropivacaine complexed with 2-hydroxypropyl- $\beta$ -cyclodextrin



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## ARTICLE INFO

## Article history:

Received 1 July 2017

Received in revised form

9 August 2017

Accepted 31 August 2017

Available online 4 September 2017

## ABSTRACT

The data presented in this article are related to the research article entitled “Probing the dynamics of complexed local anesthetics via neutron scattering spectroscopy and DFT calculations (<http://dx.doi.org/10.1016/j.ijpharm.2017.03.051>)” (Martins et al., 2017) [1]. This work shows the molecular and structural behavior of the local anesthetics (LAs) bupivacaine (BVC,  $C_{18}H_{28}N_2O$ ) and ropivacaine

DOI of original article: <http://dx.doi.org/10.1016/j.ijpharm.2017.03.051>

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<http://dx.doi.org/10.1016/j.dib.2017.08.053>

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(RVC, C<sub>17</sub>H<sub>26</sub>N<sub>2</sub>O) before and after complexation with the water-soluble oligosaccharide 2-hydroxypropyl-β-cyclodextrin (HP-β-CD).

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Specifications Table

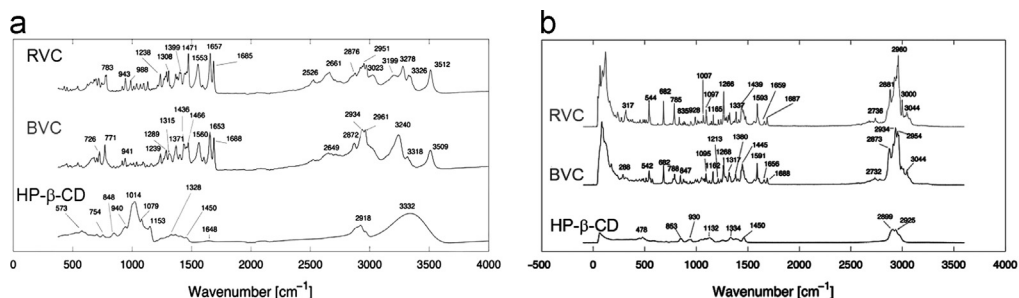
|                                 |  |
|---------------------------------|--|
| Subject area                    | Physics, chemistry and pharmaceuticals   |
| More specific sub-<br>ject area | Molecular vibration on complexed local anesthetics   |
| Type of data                    | Figures and table  |
| How data was<br>acquired        | The Raman spectroscopy (RS) data were obtained on a MultiRAM FT spectrometer, Bruker, equipped with a Nd:YAG laser. The Fourier transformed infrared spectroscopy (FTIR) data was acquired on an ATR Crystal spectrometer, Bruker. The X-ray diffraction (XRD) data was collected on a D8 – Discover diffractometer, Bruker. |
| Data format                     | Raw and analysed data  |
| Experimental<br>factors         | Powder samples   |
| Experimental<br>features        | RS was collected between 200 and 3500 cm <sup>-1</sup> with an incident wavelength of 1064 nm and a laser powers of 250 mW. FTIR data were collected between 400 and 4000 cm <sup>-1</sup> with 500 scans for each sample. XRD data was collected with a Cu radiation source.  |
| Data source<br>location         | Copenhagen, Denmark.   |
| Data accessibility              | Data are available in this article.  |
| Related research<br>article     | Probing the dynamics of complexed local anesthetics via neutron scattering spectroscopy and DFT calculations   |

Value of the data

- Relevant data on the characterization of local anesthetics RVC and BVC and the respective complexes.
- Data to be used on understanding molecular changes on local anesthetics after complexation in HP-β-CD.
- RS, FTIR and XRD data to be used as complementary information to several characterization techniques on pharmaceutical research.

1. Data

FTIR and Raman spectra for BVC, RVC and HP-β-CD are presented in Fig. 1 (a) and (b) to be used as complementary data for the neutron scattering analysis presented on reference [1]. Table 1 presents the modes assignment, based on references [2–4]. Fig. 2(a) presents FTIR data for BVC and RVC BVC after complexation with HP-β-CD, thus BVC-HP-β-CD and RVC-HP-β-CD. Fig. 2(b) shows the respective RS spectra. In Fig. 3, X-ray diffraction data is presented for BVC-HP-β-CD and RVC-HP-β-CD, i.e. RVC after complexation with HP-β-CD.



**Fig. 1.** (a) FTIR and (b) Raman spectra of RVC, BVC and HP-β-CD. All data were collected at room temperature.

**Table 1**

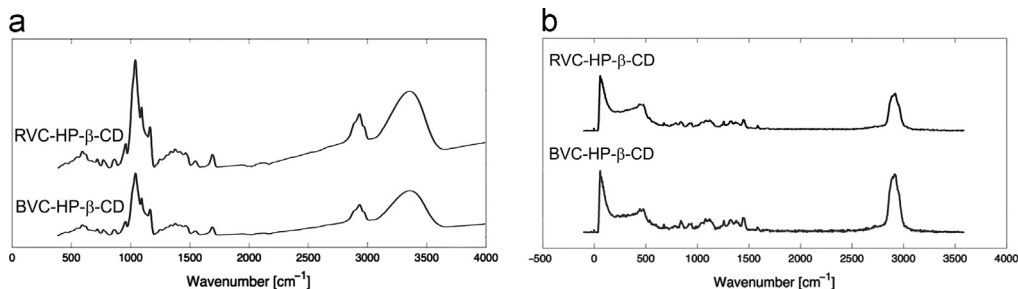
Modes assignment for FTIR and RS for BVC, RVC and HP-β-CD.

| Sample             | Frequencies (cm <sup>-1</sup> )           | Modes Assignment   |
|--------------------|---|--|
| <b>BVC and RVC</b> | • 3509 (BVC)                              | O-H bond stretching  |
|                    | • 3512 (RVC)                              |  |
|                    | • 3240 and 3318 (BVC)                     | Stretching of hydrogen-bonded N-H group of the mono-substituted amides, O=C-N-H                    |
|                    | • 3199, 3278 and 3326 (RVC)               |  |
|                    | 2960                                      | CH <sub>3</sub> stretching   |
|                    | 2500 - 2700                               | Stretching of N-H-Cl   |
|                    | 1700 - 1600                               | C=C and C=O stretching   |
|                    | 1680 - 1630                               | Amide carbonyl stretching band (♦(C=O))  |
|                    | 1550                                      | Amide II vibration (C-N stretching vibrations together with N-H bending)                           |
|                    | 1250                                      | C-N-H stretch vibrations   |
| <b>HP-β-CD</b>     | 1470 - 1250                               | Information on the rings and the methyl (CH <sub>3</sub> ) and methylene (CH <sub>2</sub> ) groups |
|                    | 1466, 1436 and 1471                       | CH <sub>2</sub> -bending   |
|                    | • 1371 (BVC)                              | CH <sub>3</sub> bending  |
|                    | • 1399 (RVC)                              |  |
|                    | 1000 - 600                                | Bending of C-H groups located either in the rings or in the carbon groups                          |
|                    | Around 780                                | Adjacent CH wag modes  |
|                    | 3332                                      | O-H bond vibration   |
|                    | 2928                                      | C-H out of phase stretching  |
|                    | 1450 and 1328                             | C-H bending  |
|                    | 1153, 1079 and 1014 vibrations below 1000 | C-O stretching<br>Different types of bending of C-H bonds in the aromatic ring.                    |

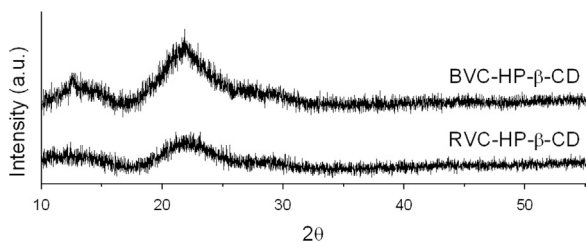
## 2. Experimental design, materials and methods

### 2.1. Materials

BVC hydrochloride monohydrate in the form of racemate (BVC.HCl, C<sub>18</sub>H<sub>28</sub>N<sub>2</sub>O.HCl.H<sub>2</sub>O) and RVC hydrochloride monohydrate (RVC.HCl, C<sub>17</sub>H<sub>26</sub>N<sub>2</sub>O.HCl.H<sub>2</sub>O) were donated by Cristália Prod. Quím. Farm. Ltda (Itapira, SP, Brazil). 2-hydroxypropyl-β-cyclodextrin, HP-β-CD, (Kleptose HP®) was obtained from Roquette Serv. Tech. Lab. (Lestrem, Cedex, France). Deionized water (Elga Maxima System, Elga, High Wycombe, UK) was used throughout the experiments. All other reagents were of analytical grade.



**Fig. 2.** FTIR(a) and RS(b) data for RVC and BVC after complexation with HP- $\beta$ -CD, thus RVC-HP- $\beta$ -CD and BVC-HP- $\beta$ -CD.



**Fig. 3.** X-ray diffraction data for BVC after complexation with HP- $\beta$ -CD (BVC-HP- $\beta$ -CD) and for RVC after complexation with HP- $\beta$ -CD (RVC-HP- $\beta$ -CD). The data were collected with Cu radiation ( $\lambda = 1.54 \text{ \AA}$ ). A baseline was subtracted from the data for background correction and BVC-HP- $\beta$ -CD data was shifted for better visualization.

## 2.2. Sample preparation

Samples were prepared as described in [5]. Inclusion complexes were prepared by stirring equimolar amounts of the local anesthetics (racemate BVC.HCl and RVC.HCl) and HP- $\beta$ -CD (1:1 M ratio) in deionized water at room temperature ( $25 \pm 1$ ) °C for 24 h. After completely dissolution and reaching equilibrium (4 h), the solution was freeze-dried (Labconco-freeze dry system/Freezone® 4.5) and stored at -20 °C until further use.

## 2.3. Fourier Transformed Infrared Spectroscopy (FTIR)

FTIR spectra were collected to all samples at room temperature between 400 and 4000  $\text{cm}^{-1}$ , using an ATR Crystal from Bruker. For each sample 500 scans were carried out. A background measurement was collected at the beginning of the experiment, and the obtained signal was subsequently subtracted from all the other measurements.

## 2.4. Raman Scattering (RS)

RS between 200 and 3500  $\text{cm}^{-1}$  were collected at room temperature using a MultiRAM FT-Raman spectrometer from Bruker equipped with a Nd:YAG laser. An incident wavelength of 1064 nm was used to measure the powder samples that were carefully mounted inside of glass vials. The powder samples were put in small glasses and a laser power of 250 mW was used to measure the HP- $\beta$ -CD and the LA samples. Due to the lower density of the complex BVC-HP- $\beta$ -CD, very thin pellets were made with the encapsulated drugs powders and a laser power of 500 mW was used. The data analysis was only qualitative in this experiment.

## 2.5. X-ray diffraction

BVC and RVC after complexation with HP- $\beta$ -CD were investigated by X-ray powder diffraction (XPD) in a Bruker – D8 Discover diffractometer (Cu radiation –  $\lambda = 1.54 \text{ \AA}$ ). The experiments were conducted with a  $0.01^\circ$  step, between  $10^\circ$  and  $55^\circ$ . A baseline was subtracted from the data for background correction and *BVC-HP- $\beta$ -CD data was shifted for better visualization.*

## Acknowledgements

Work by MLM was financed by the Science without Borders Program (Grant number 205609/2014-7) and HJ partially funded by an internship grant offered by the Institute Laue-von-Langevin (ILL). ECS work was financed by the Norwegian Research Council (RCN) SYNKØYT Program (project number 228551). The work of RI was part of a student project and supported by collaboration between HNB and AM. EP acknowledges FAPESP (#14/1447-5) grant. We acknowledge the support of the ILL in providing the neutron research facilities used in this work. This was financed by NMI3, CoNext and Danscatt. The thermoanalysis apparatus used in the work were financed by Carlsberg-fondets (grants 2013\_01\_0589 and CF14-0230). This research also used resources of the National Energy Research Scientific Computing Center, a DOE Office of Science User Facility supported by the Office of Science of the U.S. Department of Energy under Contract No. DE-AC02-05CH11231. JE would also like to thank the Physics and Chemistry of Materials Group (T-1) at LANL for making computing resources available. HNB thanks Stéphane Rols (ILL) for fruitful discussions concerning the analysis of the quasi-elastic data. We also thank Niels Vissing Holst for technical support on X-ray diffraction data collection.

## Transparency document. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.dib.2017.08.053](https://doi.org/10.1016/j.dib.2017.08.053).

## Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.dib.2017.08.053](https://doi.org/10.1016/j.dib.2017.08.053).

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